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FOREWORD

This report was prepared by the University of California, under USAF Contract No. AF 33(616)-7763. This contract was initiated under Project No. 7350 "Ceramic And Cermet Materials Development," Task No. 735001, "Non-Graphitic." The work was administered under the direction of the Metals and Ceramics Laboratory, Directorate of Materials and Processes, Aeronautical Systems Division, with Mr. J. D. Latva and Dr. C. T. Lynch acting as project engineers.

This report covers work conducted fro..1 February 1961 to July 1962.

ABSTRACT

This summary report presents the progress completed on the investigation of the mixing and sintering characteristics of Al_2O_3 and MgO particles. Separate inter-related areas of research have been investigated which include particle preparation, dry and wet mixing, and the sintering behavior of Al_2O_3 and MgO.

The techniques of particle reduction and analysis of particle size distribution have been investigated by determining the variables which affect the resulting particles. Emphasis has been placed on control conditions for a jet pulverizer.

Variables encountered in the dry mixing operation have been found to be the mixer design, loading methods, and sampling methods. Analysis of these variables has been made by determining the standard deviation from a statistical number of samples. Surface properties of Al_2O_3 and MgO in aqueous media have been investigated in order to understand wet mixing behavior. Determinations of the nature of the electrical double layer of the particles have been made.

The basic mechanisms and the characteristics of the reaction between MgO and Al_2O_3 have been studied with single crystals. The understanding of the fundamentals of the reaction has enabled the establishment of optimum sintering conditions. Some of the properties of sintered compacts due to varied sintering conditions have been determined.

This technical documentary report has been reviewed and is approved.

W. G. RAMKE
Chief, Ceramics and Graphite Branch
Metals and Ceramics Laboratory
Directorate of Materials and Processes

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INTRODUCTION

This report is a summary of progress in research on the dependence on the structure of ceramic bodies, and in turn their properties, on the mixing and sintering of raw material particles. The basic system used in this research is alumina and magnesia which react to form spinel during the sintering process. Research has been carried out on particle preparation, dry mixing phenomena, surface factors that affect wet mixing, sintering of mixtures of particles, and basic studies of the sintering mechanism.

The following material summarizes initial progress that has been made toward the overall objectives of the research program. The work is continuing.

PARTICLE PREPARATION

Particle preparation consists of particle reduction and analysis of particle size distribution. Particle reduction has been undertaken with both a two-inch and a four-inch jet pulverizer. The smaller pulverizer has proven to be totally unsatisfactory and the larger pulverizer has given only a limited amount of success. The choice of using a jet pulverizer had been made on the basis of its reputed high efficiency in the production of fine particle sizes and its ability to minimize impurity pick-up in the grinding process. However, the extremely hard materials used in this project cause excessive wear of the pulverizer and the introduction of a high percentage of iron contamination. This problem has been partly alleviated by modifying the pulverizer with rubber or nylon linings on abrasion surfaces.

Tests to establish standard control conditions for the jet pulverizer have been made with quartz so that results can be compared directly with the known grinding characteristics of quartz. Size fractions of 10 x 14, 14 x 20, and 20 x 28 mesh were passed through the pulverizer at a feed rate of 500 grams per hour. The particle size distribution that resulted from each fraction appeared to be approximately the same within the scatter, and thus, the feed size range does not appear to be a variable. The Schuhmann plots for these results are presented in Figure 1. The slopes from these plots are:

$$10 \times 14 \text{ mesh } - \alpha = 2.25$$
$$14 \times 20 \text{ mesh } - \alpha = 2.03$$
$$20 \times 28 \text{ mesh } - \alpha = 2.36$$

The established slope for quartz from ball mill and rod mill wet grinding has been determined as 0.90. The discrepancy is believed to be due to agglomeration of the dry quartz particles which may not break up during measurement. This effect may also be responsible for the wide scatter observed. Tests are being conducted to determine whether this possibility can be substantiated.

Changes in the feed rate from 500 g/hr to 1500 g/hr, 2500 g/hr and 3500 g/hr have created a successively higher proportion of coarse material. The

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following results from a 10 x 14 mesh series is typical of this behavior:

Feed rate (g/hr)	500	1500	2500	3500
50% finer (μ)	6.5	8	9	10.2

The decrease in the efficiency in producing fines with increased feed rate is believed to be due to short circuiting through the pulverizer. The requirement for fine particle sizes appears to limit the operation of the jet pulverizer to low feed rates.

Grinding has also been done in alumina ball mills. Particle size distributions of Al_2O_3 using alumina balls have given reproducible results and a sufficient proportion of fines. MgO particles have been successfully ground using large single crystals of MgO as the grinding media. Determination of size range and reproducibility is being undertaken.

All particle size distributions have been determined using a continuously recording sedimentation balance. Standardization tests with micron sieves and the optical microscope have shown that accurate results are obtained with the balance.

DRY MIXING

The mixing of materials has been considered an industrial process and, therefore, a fundamental approach to mixing has long been neglected. An attempt is being made to determine the variables that affect mixing and a parameter to define the degree of mixing.

The selection of a mixing criterion must be related to the property of the materials being analyzed. In this case, the amount of spinel formed in sintering is the analyzed property and the area of particle contact would be the ideal mixing parameter. Attempts are being made to experimentally measure this contact area but an easier and more immediate parameter which is being used is the standard deviation of the relative concentration from the mean of a number of samples.

The analytical procedure involved in the determination of magnesia and alumina in a mix-sample is difficult and expensive. Because a great number of samples are necessary for the statistical analysis, preliminary investigations have been carried out using silica and $CaCO_3$ mixtures which can be analyzed by simple titration. The preliminary investigations have been concerned with the characteristics of the mixing method and not with the properties of the mixing particles.

Mixing has been done in a laboratory twin shell blender and a rotary drum blender. The twin shell blender has a capacity of 710 cc in which the solids occupy 14 percent of the total mixing volume. The rotary drum blender has a capacity of 480 cc in which 34 percent of the mixing volume is occupied by solids. Both blenders are operated at 24 revolutions per minute.

The effect of mixer design is illustrated in Figure 2. Although the initial areas of contact are approximately the same for both blenders, the rotary drum blender appears to have the greater efficiency for a total number of revolutions less than 72, while for a greater number of revolutions it appears to be less efficient than the twin shell blender. It appears that the mixer design incorporates the three mixing mechanisms (diffusion, shear, and convection) to a different degree.

Mixing curves are plots of the average standard deviation among the series of samples taken from the mixer at different mixing times. The samples are sufficiently small as not to alter the contents of the blender as a whole (about 0.1 percent of the whole). A typical mixing rate curve indicating the spread of standard deviations is shown in Figure 3 for the twin shell blender It can be seen that the curve is not changed appreciably if the limits of the standard deviation are used.

Two methods of loading the twin shell blender have been used (end and layer) and are illustrated schematically in Figure 4. The effect created by different loading methods is presented in Figure 5, which shows the change in the mixing rate curve. It is apparent that consistent loading procedures must be adhered to in order to reproduce the mixing rates.

The two sampling methods used (end and axial) are also schematically illustrated in Figure 4, and the results of the sampling methods are presented in Figure 5. The effect demonstrates that an apparent degree of mixing may be determined solely from the method of sampling. It is believed that this effect is caused from an agglomeration of particles which subsequently break up.

The standard deviation within the sample has also been investigated. The standard deviation obtained from the normal sampling method was 8.6. Larger samples were chosen and the standard deviation from six sub-samples within each was 2.0 while the standard deviation of their means was 8.1. Although the samples had a higher standard deviation, their mean value was more representative of the whole than a single larger sample. These results indicate that material to be used for sintering must be selected from the entire batch if it is expected to be representative of the batch.

The effect of size distribution on mixing was studied by placing two materials side by side in a rotary drum blender and obtaining size distributions at various levels after mixing. Each of the materials had its own characteristic size distribution. It was found that diffusion was wholly operating and that size distribution did not influence mixing.

Some of the variables which affect mixing or the analysis of mixing have been determined in the preliminary investigation conducted. It is apparent that the control of these variables is necessary to produce predictable mixing parameters. The effect of changes in these variables on the mixing parameter must also be studied.

SURFACE PROPERTIES OF Al_2O_3 AND MgO IN AQUEOUS MEDIA

The objective of tł ... part of the study is to determine the correlation between the electrical double layer at the particle-solution interface and wet mixing behavior. Streaming potential studies of Al_2O_3 and MgO were conducted in order to provide information on the electrical double layer. The electrical double layer controls the stability of a suspension of solid particles of a single type, that is, the presence or absence of repulsion due to interaction of the double layers determines whether the suspension is flocculated or dispersed. In a mixture of particles of two kinds the state of a suspension will depend upon the presence or absence of a double layer on one or both kinds of particles, and on the relative signs and

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magnitudes of the double layers. Hence, if it is known how the surface potentials of Al_2O_3 and MgO vary with pH and ionic strength and content it should be possible to correlate wet mixing behavior with the composition of the mixing liquor.

The corundum used for this study was obtained in the form of synthetic sapphire boules from Linde Company. The magnesia was Norton Company's optical grade magnorite. The large single crystals of alumina and magnesia were crushed by hand in a small jaw crusher, and each material separated by screening into two size fractions, 28/35 mesh and 48/65 mesh. Particles of iron introduced in crushing were removed with a hand magnet. The corundum samples were leached in hot concentrated HCl to remove the last traces of iron, followed by rinsing with distilled water until the rinse water was free of chloride ion. The corundum was then stored under distilled water in covered Pyrex beakers. Because of its relatively high solubility and its tendency to hydrate, the magnesia was stored dry in Pyrex beakers placed inside a dessicator. Samples to be used were taken out individually, leached in hot 1N HCl and rinsed with distilled water until the rinse water was free of chloride ion.

To prepare highly pure conductivity water, free from electrolytes and surface active agents, a fused silica double distillation apparatus was used, and the product collected in a CO_2-free atmosphere. All water used had a specific conductivity of less than 6×10^{-7} ohm^{-1} cm^{-1}. Initially, a commercial ion exchanger was used, giving water with specific conductivity of less than 4×10^{-7} ohm^{-1} cm^{-1} (theoretical conductivity of pure water is $.55 \times 10^{-7}$ ohm^{-1} cm^{-1}), but experiments conducted after the double-still was received showed that water from the ion exchanger was changing the apparent isoelectric points of the solids, possibly because of neutral organic molecules introduced into the water by the resin.[1]

All chemicals used were reagent grade. HCl and NaOH were prepared from standard ampoules obtained from Bio-Rad Laboratories, and have been stored and dispensed in CO_2-free atmospheres.

The apparatus used in this study consists of the cell assembly and flow system described previously by Fuerstenau.[2] The electrical measuring equipment has been modified (see Figure 6) to include a multi-range recorder to facilitate the measurement of rapidly changing potentials.

The measurement of the specific conductance of the solution has been simplified from the methods of previous investigators by the use of precision resistors, selected to be approximately equal to the resistance encountered in the test solutions. The actual solution resistance in the streaming potential cell is found from the relation

$$R_1 = (\frac{E_1 - E_2}{E_2}) R_2$$

where R_1 is the solution resistance in streaming potential cell, R_2 is the known resistance, E_1 is the actual streaming potential, and E_2 is the streaming potential measured with R_2 connected in parallel with streaming potential cell. For a given specific conductivity, the zeta potential is directly proportional to the streaming potential.

In early work it was found that the zero point of charge of corundum was in the region of pH 8. However, a lack of reproducibility of the data, especially near

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the zero point of charge, led to a search for some possible source of contamination of the reagent solutions. As previously mentioned, on the arrival of the double-still the conductivity water from the ion exchange resin was found to be the cause of the wayward results. After a thorough cleaning of all equipment which had been in contact with the contaminated water and preparation of new reagent solutions · with uncontaminated water, the zero point of charge of corundum was redetermined. For two successive new samples of corundum, the zero point of charge has been found to be pH 9.1 - 9.2, with good reproducibility of data between the samples (see Figure 7).

It has been found, however, that when a sample of corundum is retained in the streaming potential cell after the initial run, its apparent zero point of charge shifts to a lower pH in subsequent runs (see Figure 8). This phenomenon, if it has been observed by other investigators, has apparently been ignored. Modi,[3] for example, obtained a zero point of charge of pH 9.4, but it is known that his experimental method consisted of the use of a new sample after each run. Theoretical calculations place the estimated zero point of charge of corundum between pH 7 and pH 8.[4] This situation suggests that the observed shift in the apparent zero point of charge has two possible causes: 1) irreversible adsorption of ions is taking place at the solid surface; or 2) under the influence of the reagents, the surface is approaching some equilibrium hydrated condition.

O'Conner et al.[5] reported that the zeta potential of corundum in water could be made to change from positive to negative by annealing the samples at temperatures of 1000°C or above. They also found that the zeta potential of the annealed samples in water changed with time of aging in water after being annealed. These findings suggest that the problem of the changing apparent zero point of charge may be one of reaction kinetics, i.e., of a slowly attained equilibrium. In this case, the zero point of charge of a sample will depend upon the prior history of the surface.

The following experiments are being conducted to obtain information on the factors which cause the zero point of charge to vary: 1) measuring the variation of the zero point of charge with repeated cycling of the pH of the solution, i.e., determining the existence of hysteresis effects or irreversible changes; and 2) measuring the variation of the zero point of charge with annealing temperature, and with aging time of the annealed samples.

Although no streaming potential measurements have been made on MgO since the rectification of the conductivity water problem, some useful conclusions can be drawn from previous results. It is certain that the zero point of charge of MgO, if one exists at all, occurs at a high pH. Theoretical calculations predict that MgO has no zero point of charge, but experiments with Al_2O_3 suggest that under conditions where equilibrium is attained slowly or specific adsorption takes place, MgO might be induced to exhibit an apparent zero point of charge.

Because of the relatively high solubility of MgO, solutions in equilibrium with it must always have ionic strengths of 10^{-4} M or higher. Correspondingly, pure water in equilibrium with MgO will have a pH in excess of 10.

In the process of leaching the MgO particles with 1N HCl, it was found that small particles of crystalline MgO, of subsieve sizes, were carried away with the first washing. The parent particles were found not to have been reduced significantly in size. Microscopic examination of their surfaces revealed etch pits along

slip lines, suggesting that the small particles had been released from the surface by preferential etching of slip bands in regions of cross slip near the surface. The slip bands were, undoubtedly, due to plastic deformation introduced in the crushing process. In the light of the change in the zeta potential found by O'Connor et al. in annealed samples of Al_2O_3, it is possible that the strain energy introduced in crushing has a significant effect on the surface charge of both MgO and Al_2O_3 in contact with a solution.

SOLID STATE REACTION BETWEEN SINGLE CRYSTALS OF MgO AND Al_2O_3

A basic study was made of the reaction between single crystals of magnesia and alumina. This study was undertaken to determine the mechanism and phenomenological aspects of the reaction so that a better understanding of the sintering process could be obtained. The mechanism has been proposed[6] and confirmed[7] previously but for an equilibrium state. This present work covers the non-equilibrium state which is of interest in the sintering process. Some phenomenological aspects of this reaction have been reported[8] but no report had been made of the crystallographic dependence of the reaction.

Sapphire boules obtained from the Linde Company were cut parallel to particular crystallographic planes. Reaction faces were ground with SiC powders and 5μ diamond paste on specimens approximately 1/2 inch on edge and 3/16 inch thick. Single crystals of MgO obtained from the Norton Company were either cleaved or cut parallel to (100) or (111) planes. Specimens with the same approximate dimensions were polished with diamond paste on parallel faces of the crystal.

The reaction couple was made by sandwiching an MgO crystal between two Al_2O_3 crystals. The couple was introduced into a preheated vertical furnace heated with $MoSi_2$ heating elements. Runs were conducted in air at 1560°C for 250 hours.

Analysis of the crystals consisted of 1) visual observations, 2) weighing each crystal, 3) measuring the thickness of the sapphire at the four corners of both reaction interfaces, and 4) orientation of the spinel product with respect to the parent crystals using the Laue camera.

Examination of the couple after the reaction revealed that a separation always occurred at the MgO-spinel interface. The spinel grew in two layers; thickness measurements showed that the boundary between the two layers corresponded to the original interface. In many cases, a fracture appeared within the sapphire running roughly parallel to the sapphire-spinel interface. These observations are shown in Figure 9.

From the thickness measurements of the two spinel layers, it is apparent that the spinel layers grew respectively from each parent crystal. The calculated ratio of the thicknesses of the two layers is 1 : 4.75 when account is taken of the solid solution existing at the reaction temperature. The measured ratio was found to be 1 : 4.83 which is in excellent agreement with the calculated value for this type of measurement. The mechanism for the spinel formation by the counter-diffusion of cations through an oxygen framework is thus confirmed under non-equilibrium conditions.

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The surface of the spinel at the spinel-MgO interface revealed characteristic textures depending upon the method of material transport across the interface. In the center of the interface where physical contact had been made, the spinel layer was clear and transparent. In the region along the edges of the crystals where contact was not made, spinel crystallites grew with well defined faces. The transport mechanism in the contact region was by diffusion; along the edge of the crystal it was by vapor-phase transport. A transition between these two consisted of a clear surface but with pores below the surface. Transport changed from vapor-phase to diffusion when the crystallites grew large enough to make contact with the MgO interface. Examples of these textures are presented in Figure 10.

The effect of the rate of spinel growth as a function of the crystallographic orientation of the parent crystals was obtained by comparing the thickness of the spinel layer with the crystallographic plane of the reaction interfaces. The results are for crystals in which contact across the interface was complete, or nearly complete.

	A	B	C	D	E
MgO plane	(100)	(100)	(100)	(111)	(111)
Al_2O_3 plane	(0001)	$(10\bar{1}0)$	$(11\bar{2}0)$	(0001)	$(22\bar{4}3)$
Average thickness of spinel layer (mm)	0.934	0.920	0.922	0.920	0.927

The results show that the amount of spinel that forms is independent of the crystallographic orientation of the parent crystals. The controlling step may thus be the diffusion of cations through the spinel layer.

The orientation study was made by constructing a stereogram of the sapphire interface before the reaction and a similar one on the corresponding spinel layer after the reaction. In each case, regardless of the initial orientation of either crystal, the spinel grown from the Al_2O_3 always formed epitaxially with the close-packed oxygen planes matching in the two structures. This relationship can be expressed as:

	Al_2O_3	$MgAl_2O_4$
Plane	(0001)	(111)
Direction	$[10\bar{1}0]$	[110]

The same relationship was found in the MgO-grown spinel when the (0001) sapphire face was used as the interface. Isomorphic crystals have been shown to have this same epitaxial relationship.[9]

No epitaxial relationships were observed for the MgO-grown spinel for $(11\bar{2}0)$ and $(10\bar{1}0)$ sapphire interfaces when diffusion was the mechanism of transport across the MgO-spinel interface. When vapor-grown spinel was formed, however, the spinel crystallites formed so that their octahedral faces (111) were parallel with the prismatic interface of sapphire.

No epitaxial dependence of spinel was found from the MgO crystal. An experiment ("E" in the table) was conducted in an attempt to induce such a dependence but the resulting relationship was other than that which was expected and corresponded to an epitaxial growth dependent upon the Al_2O_3 crystal as has been described.

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The single crystal study has confirmed the mechanism of cation diffusion through an oxygen framework under non-equilibrium conditions. This study has also shown that the rate of growth of spinel is independent of the crystallographic orientation of the parent crystals. Epitaxial growth of spinel is dependent upon the orientation of the sapphire crystal and the method of transport across the MgO-spinel interface. No epitaxial dependence was observed for the MgO crystal.

SINTERING POWDERS OF MgO AND Al_2O_3

Compacts of equal volume ratio of magnesia and alumina particles, which have been given different degrees of mixing, were sintered by a standard heat treatment. The resulting amount of spinel formed is being used as a parameter to define the degree of mixing. A tube furnace has been constructed for the sintering process which will enable varying the time and temperature of firing under controlled conditions so that the optimum heat treatment can be determined. Heat treatment is varied by moving the specimens through the furnace at a controlled rate. Analysis of the amount of spinel in the sintered compacts is being made by a quantitative x-ray diffraction method.

The development of the quantitative x-ray diffraction method has been undertaken. The standardization of the method required the determination of all the variables encountered in the use of a diffractometer. Optimum performance conditions consisted of the use of a copper target tube, a 3° beam slip, a medium resolution soller slit, a 0.2° detector slit, a nickel filter, a proportional counter and a digital printer. Because reproducibility could be a function of the scanning rate, a number of scanning rates were used. To evaluate the reproducibility of the diffractometer data and the degree of mixing within a specimen, a parameter was found which was sensitive to minor variations in the amount of spinel. The specimen used for these runs was considered to be "thoroughly mixed". Reproducibility of the diffractometer was determined by repeating diffraction runs six times on the same area and determining the standard deviation from the mean value of the spinel parameter. The results are as follows:

| Scanning rate (deg/100 sec) | 0.02 | 0.04 | 0.13 | 0.33 |
| Reproducibility (%σ) | 0.19 | 0.15 | 0.37 | 0.41 |

Though the reproducibility does not vary much with scanning rate, the faster scanning rates were found to give less accurate values as well as less precise values.

Correlation between the x-ray parameter, f_S, which represents the relative amount of spinel and the absolute amount of spinel, is being made by means of a set of standards. The standards must be considered to be thoroughly mixed. The homogeneity of a single specimen which was considered to be thoroughly mixed was determined to have a standard deviation of 3 percent.

The determination of an optimum heat treatment for the sintering process was conducted by firing specimens at various times and temperatures. The experimental activation energy for the system was determined as 103 kcal from two sets of sintering runs. This value is in agreement with the published data.[8,10] The activation energy plot is presented in Figure 11.

The change in density of the compacts after sintering runs appears to indicate that in the temperature range of 1100° - 1200°C spinel is being formed with

an increase in specimen volume. Above this temperature range the volume does not increase linearly which may indicate that a coalescence of particles is also accompanying the formation of spinel. This effect is shown in Figure 12 and is substantiated by an observed decrease in volume with additional heat treatment.

Modulus of rupture was determined on the set of sintered compacts as a function of the firing temperature and of the relative amount of spinel. Plots of these functions are made in Figures 13 and 14. The correlation between firing temperature and strength is indefinite. An excellent correlation between the strength and the relative amount of spinel gives the expression:

$$\sigma = f_s^\alpha$$

where σ is the strength, f_s is the relative spinel parameter, and α is a factor with a value of 0.38.

The compacts from which these data were obtained were thoroughly mixed. Specimens with varying degrees of mixing have strengths less than the thoroughly mixed specimens for the same heat treatment. Correlation can thus be made between the standard set and those with less thorough mixing.

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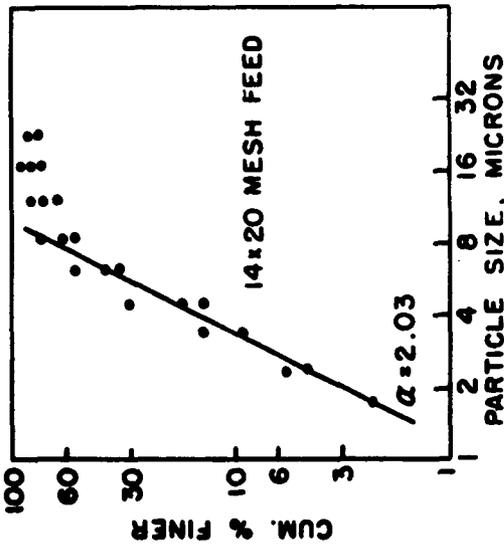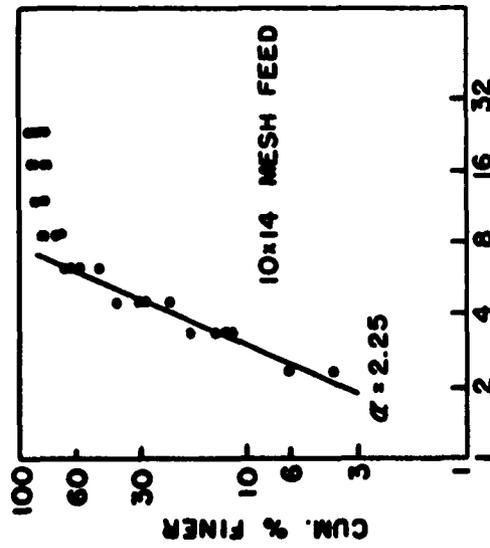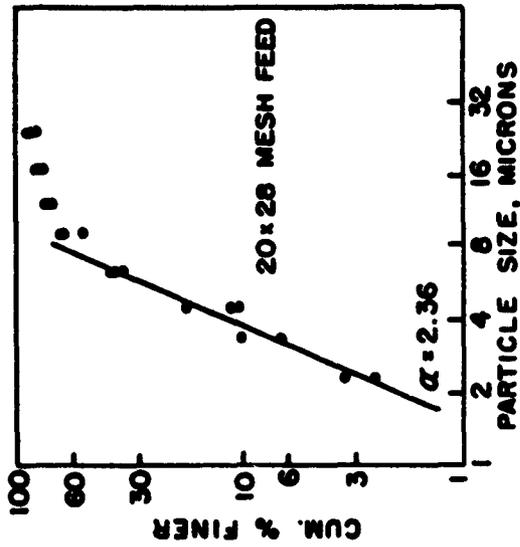
Figure 1 - Schuhmann plots of quartz ground at 500 g/hr.

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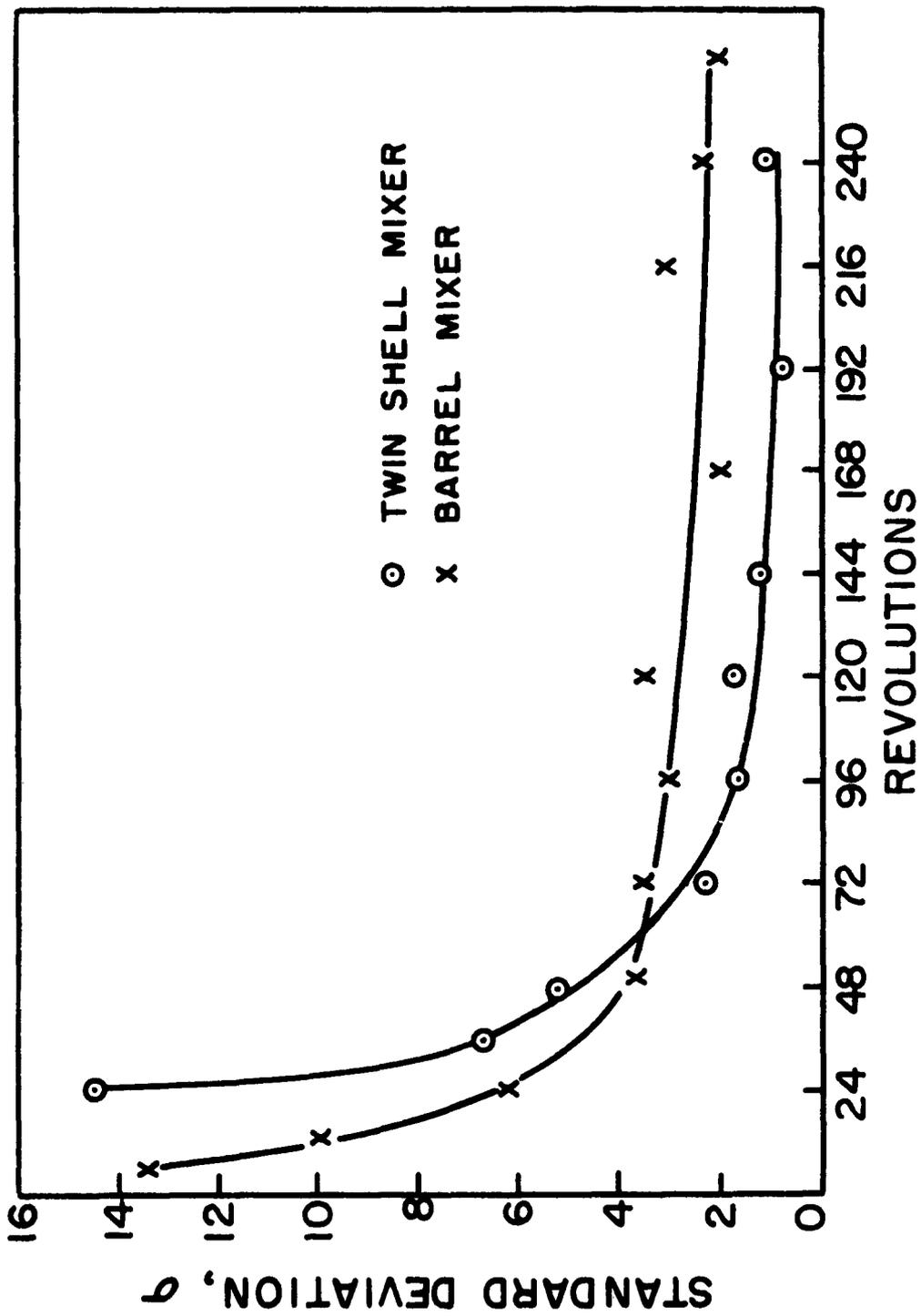
Figure 2 - Standard deviation vs. number of revolutions.

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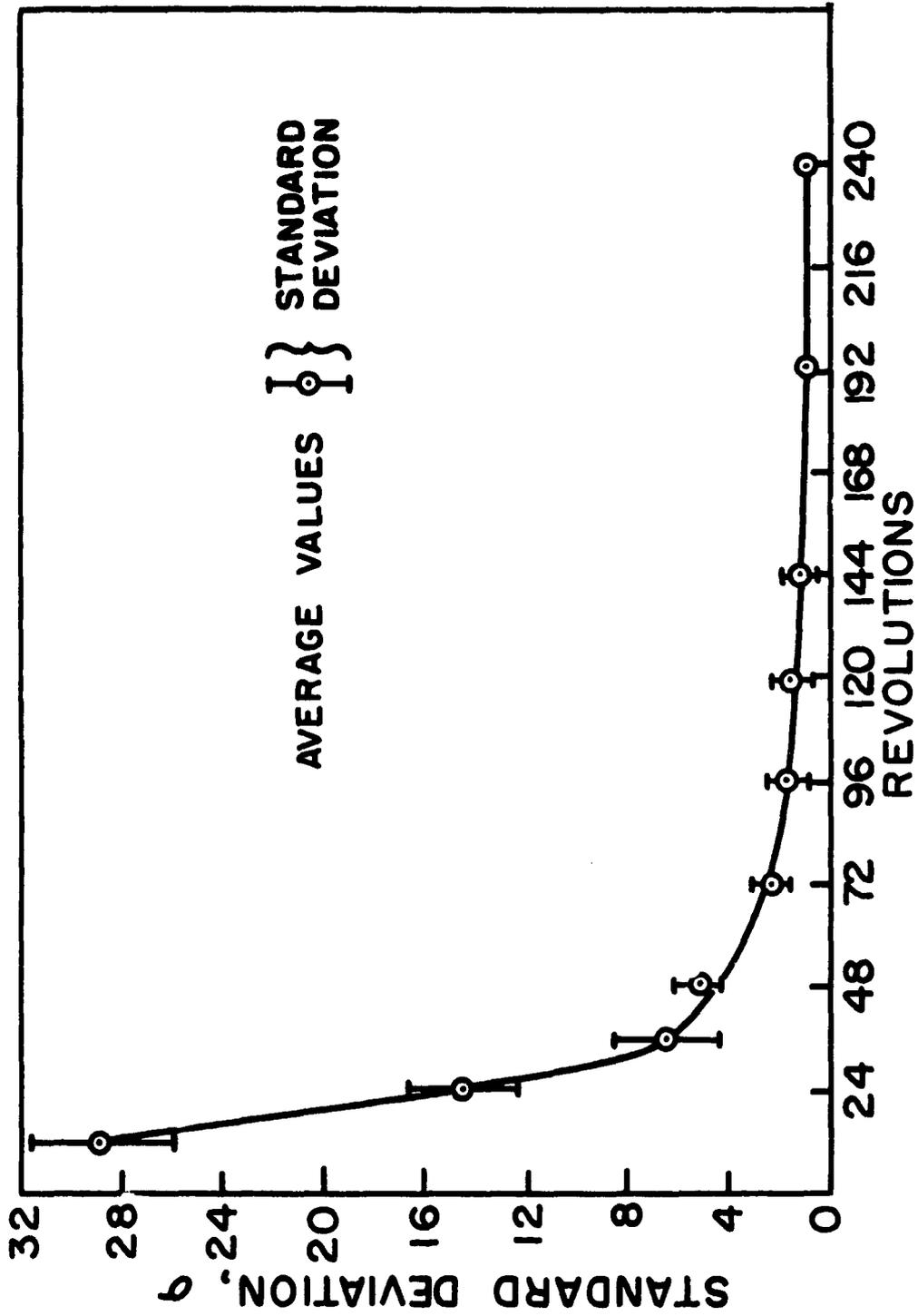
Figure 3 - Standard deviation vs. number of revolutions. Runs 5, 7-10

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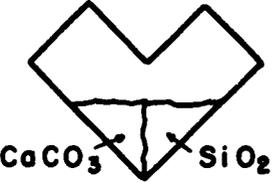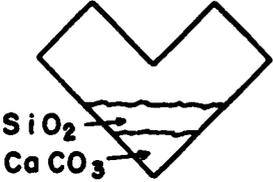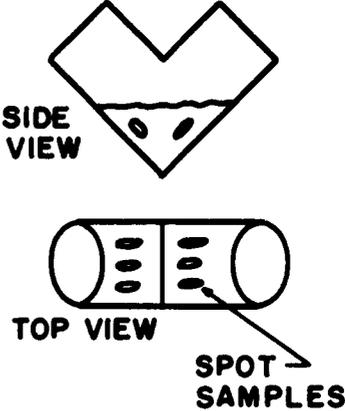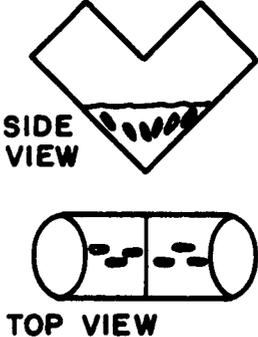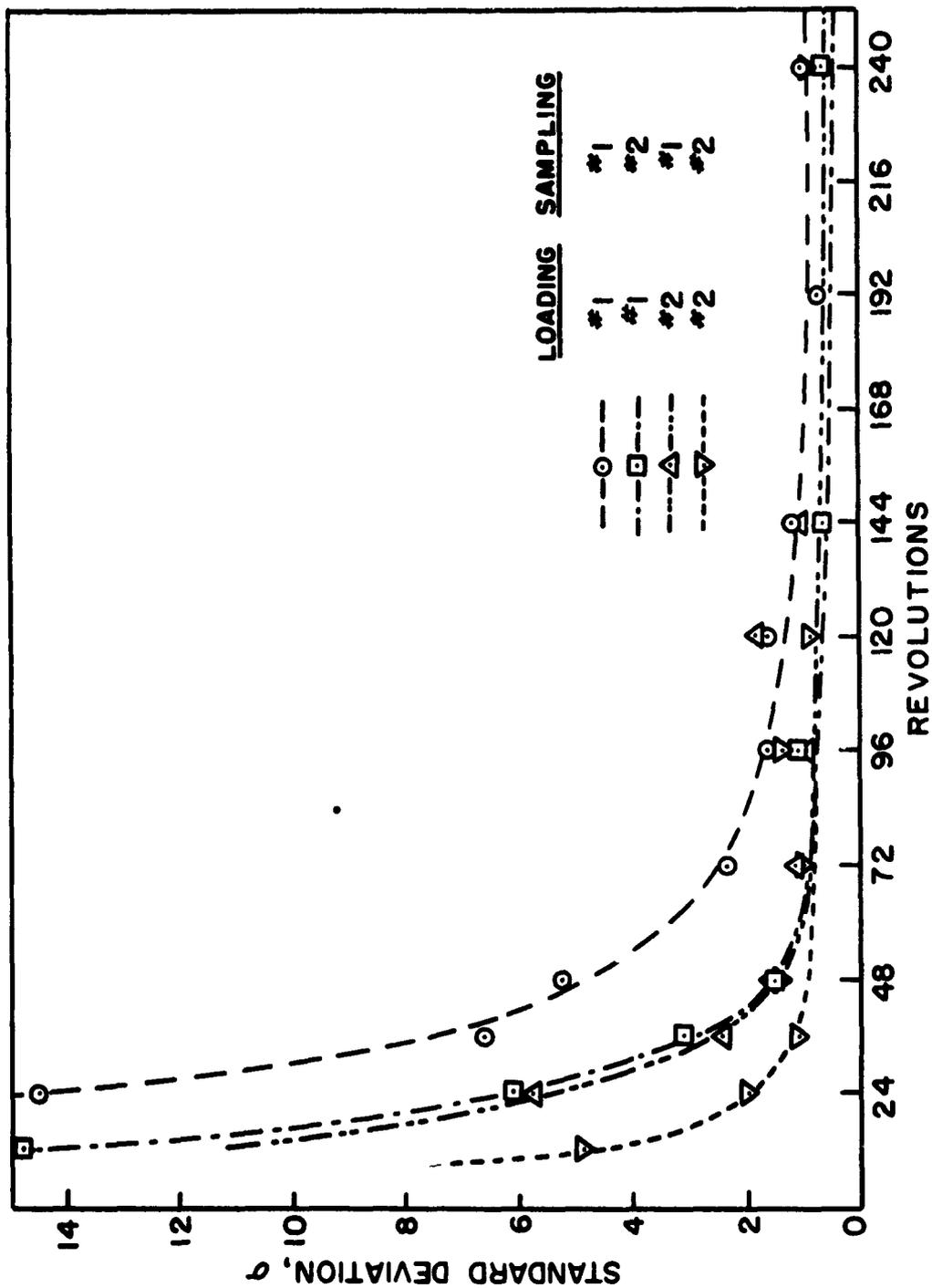
Figure 4

Figure 5 - Standard deviation vs. revolution (averages of runs).

V - (Twin hell) mixer

50-50 mix ($CaCO_3$ - SiO_2)

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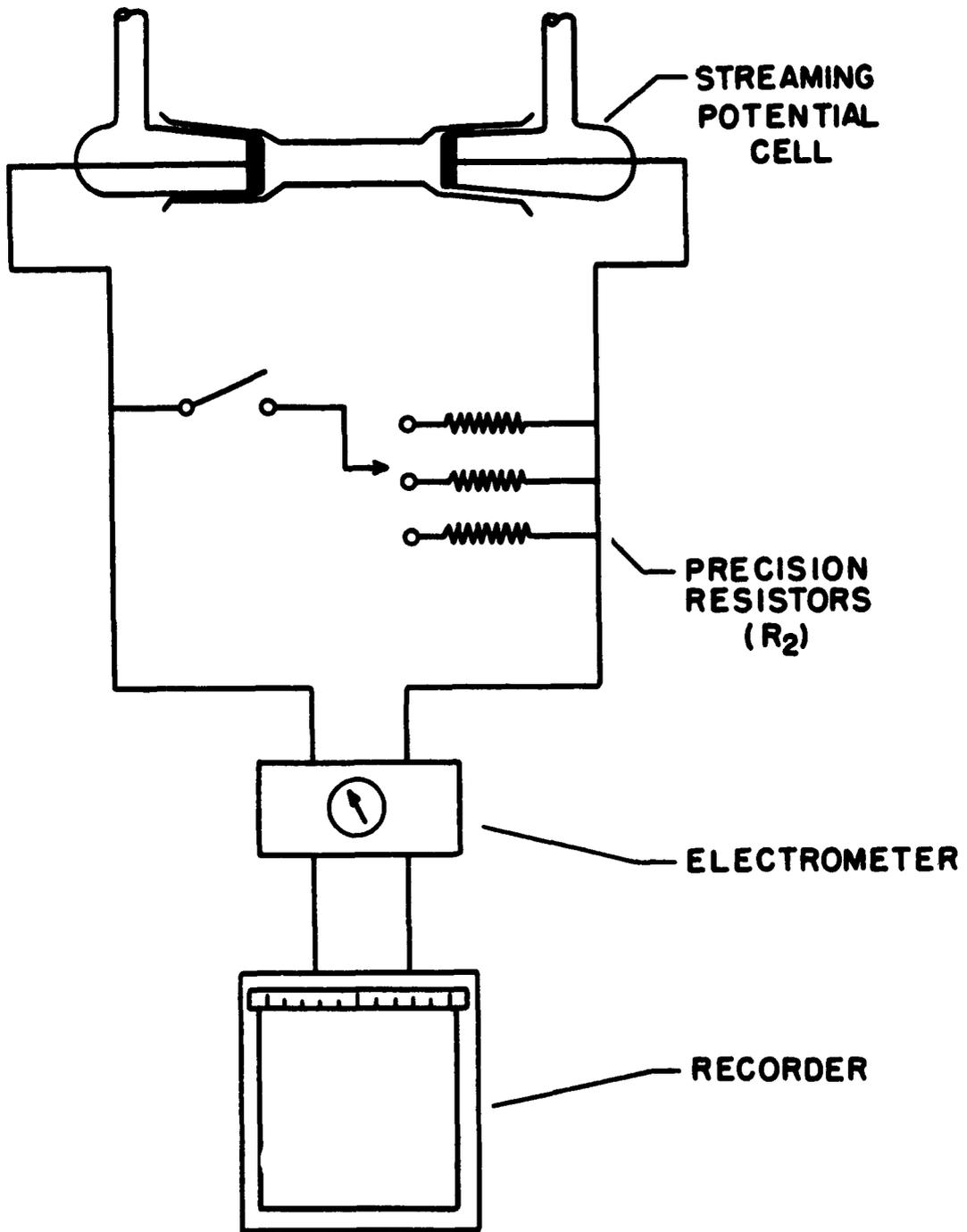
Figure 6 - Modified circuit for streaming potential measurement.
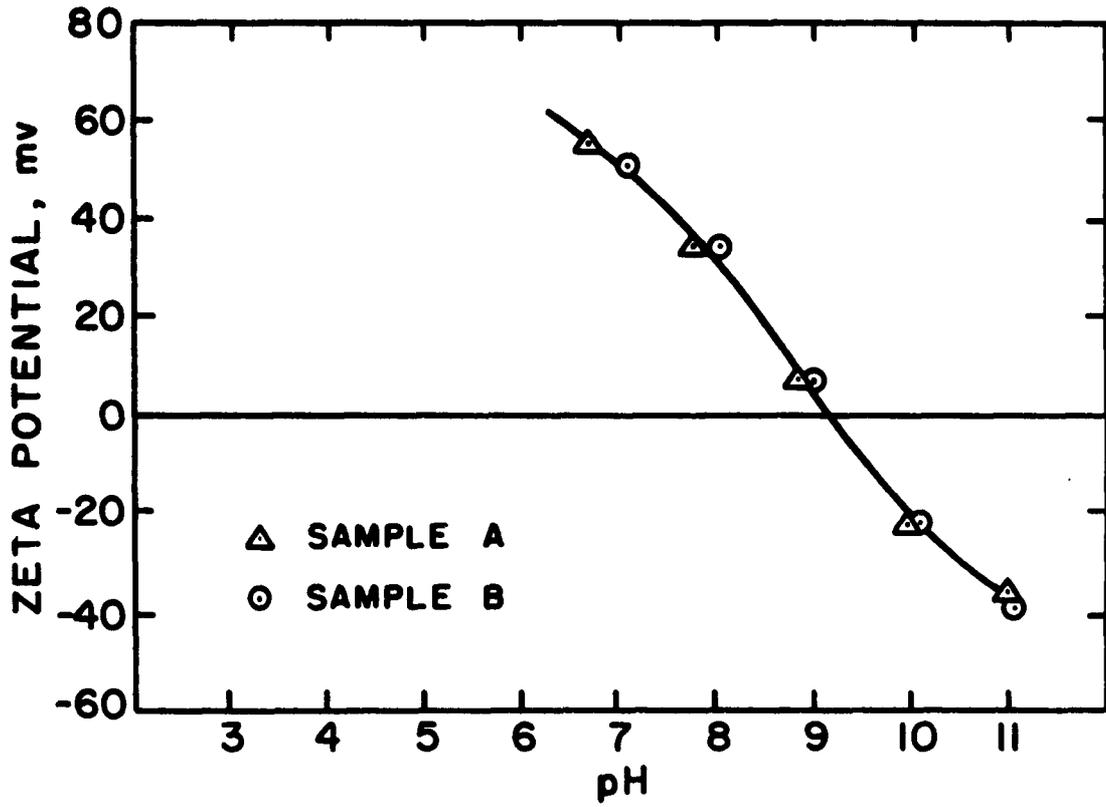
Figure 7 - Zeta potential of alumina as a function of pH.
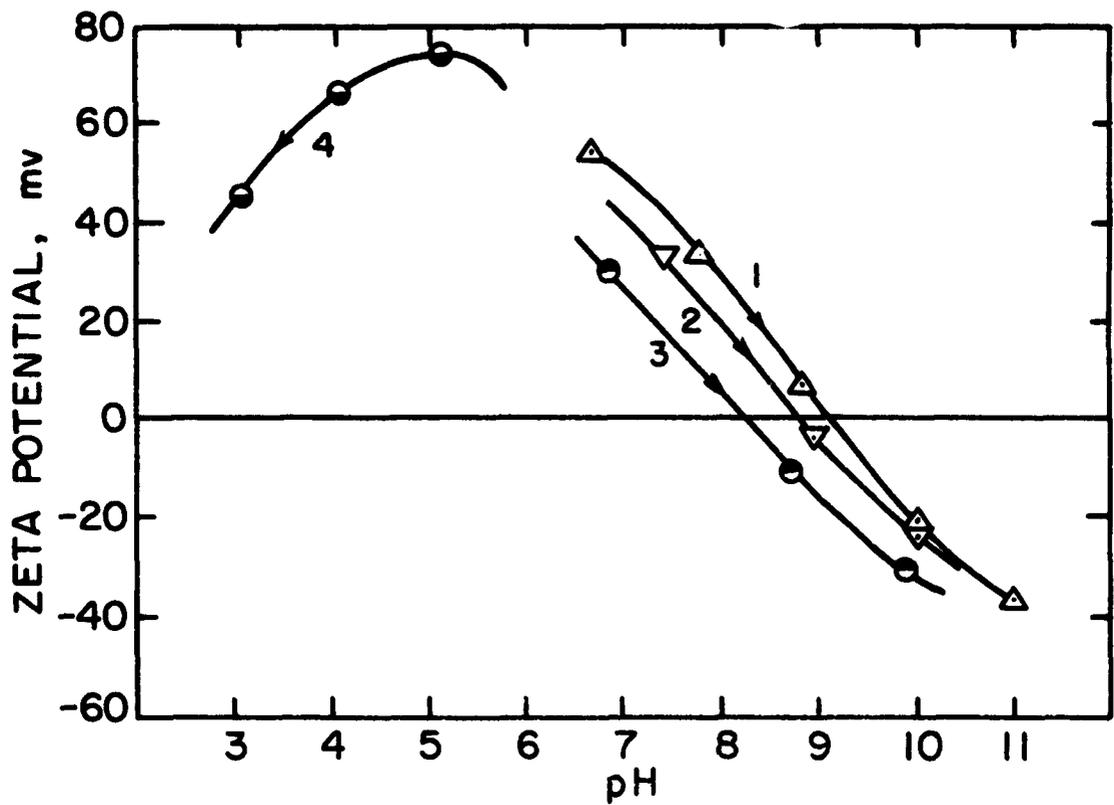
Figure 8 - Zeta potential of alumina as a function of pH. (Sample B)
Numbers and arrows indicate sequence of runs.

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Figure 9 - Section perpendicular to the interface showing two spinel layers and the fracture in sapphire (40 X).

(a)   (b)

(c)   (d)
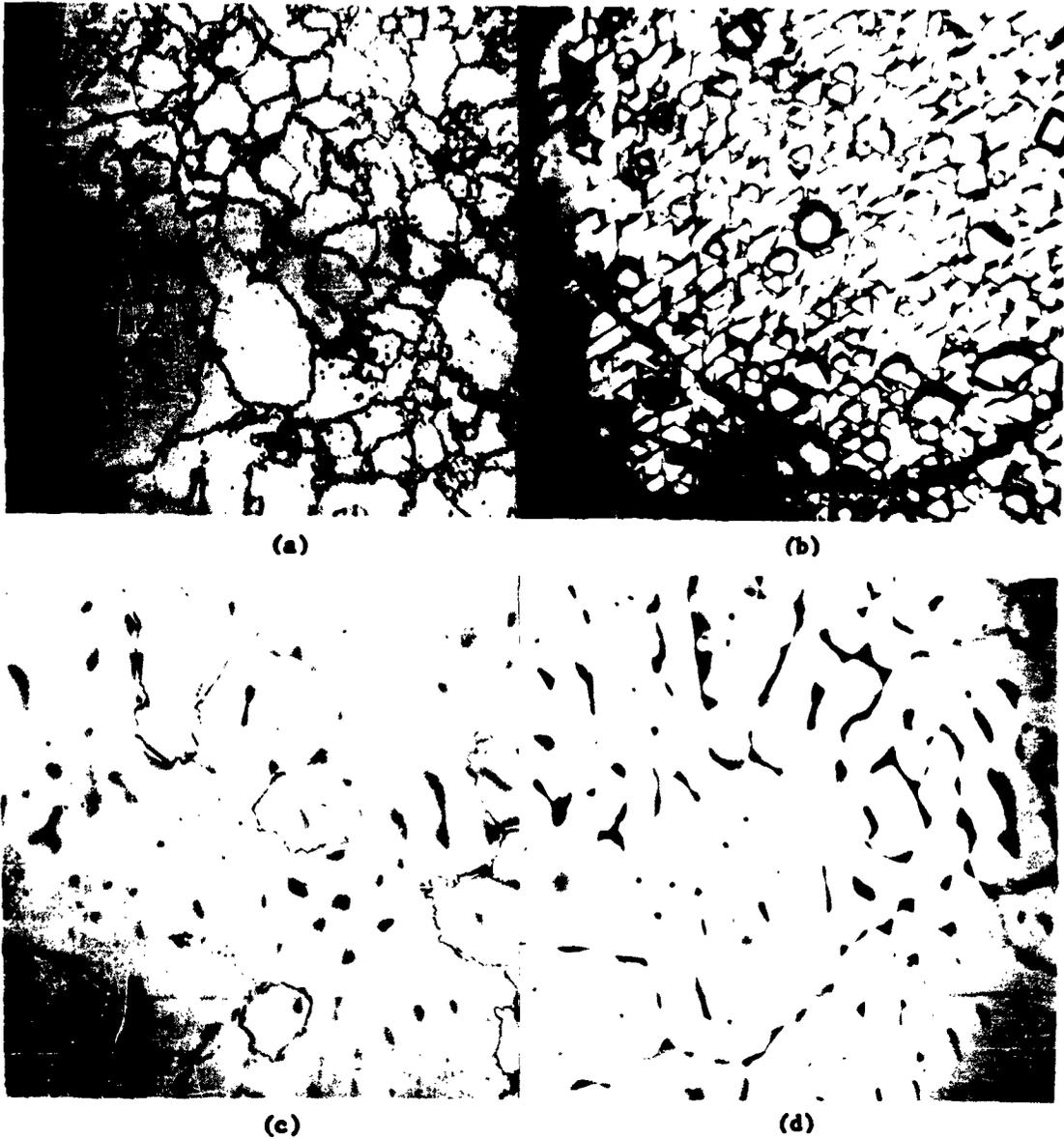
Figure 10 -   a) Grain boundaries in clear region (130 X);
              b) Crystallites in vapor-grown region (40 X);
              c) Surface of the transition region (130 X);
              d) Pores below surface, same as c) (130 X).
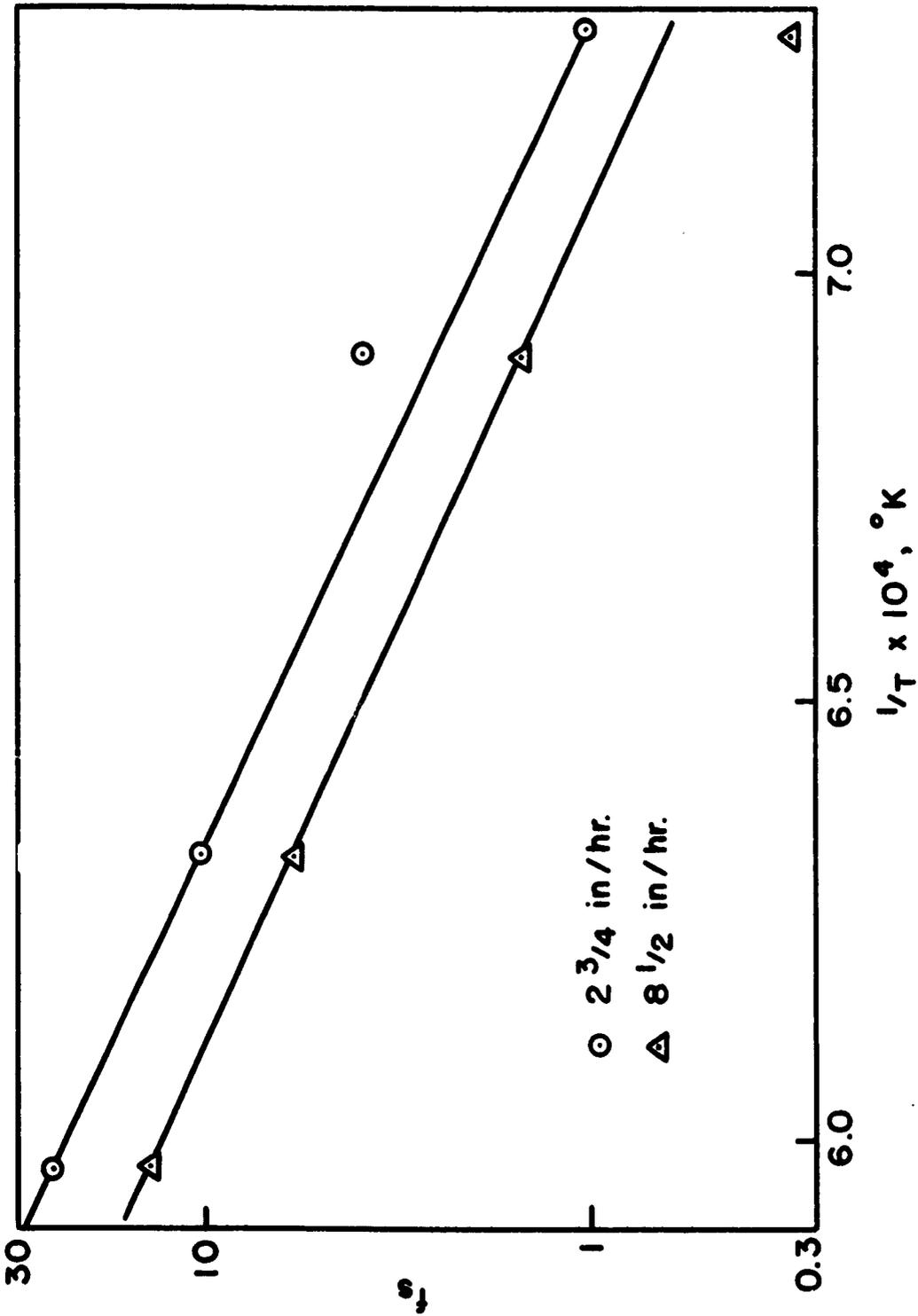
Figure 11 - Relative amount of spinel vs. reciprocal temperature of firing.
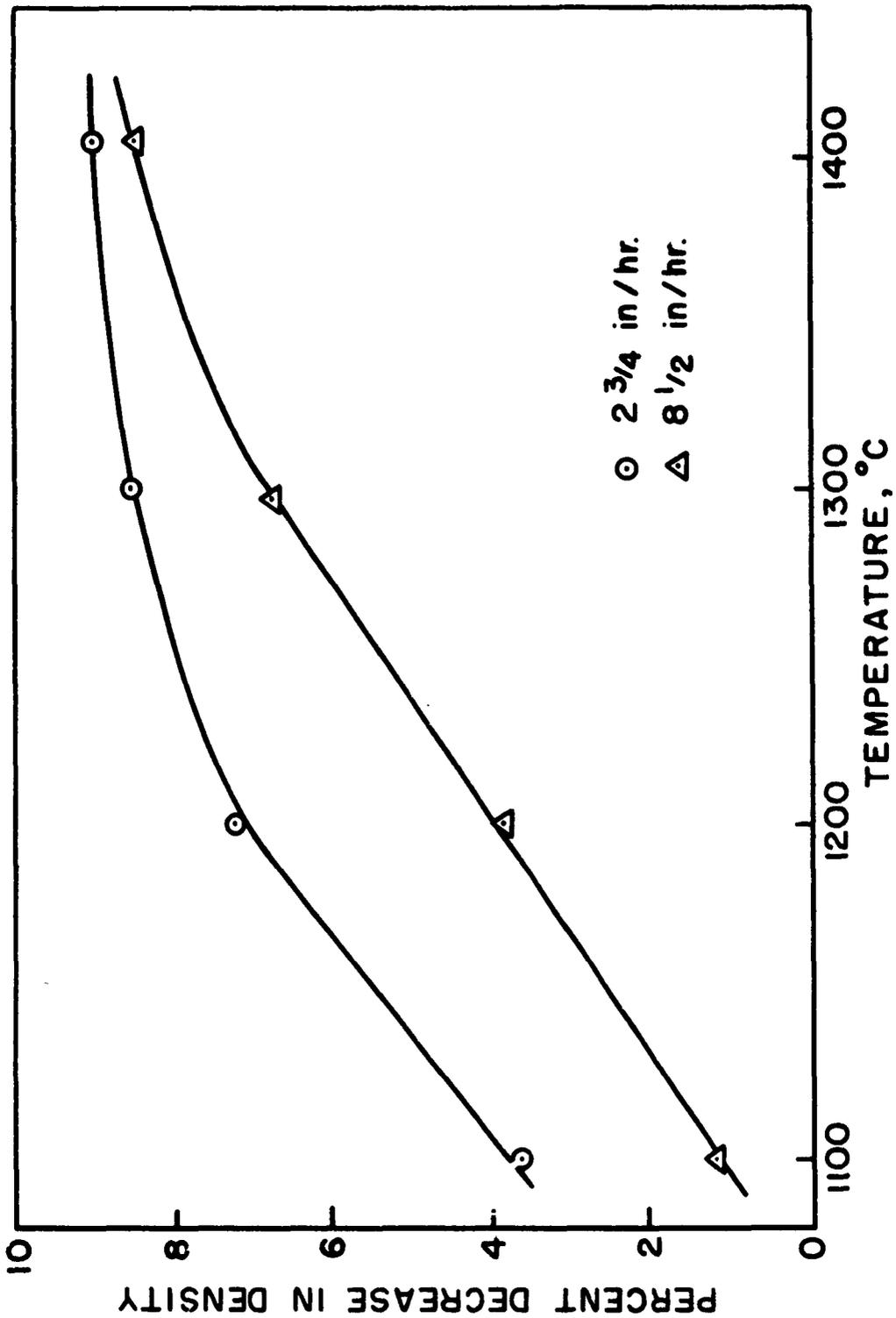
Figure 12 - Percent decrease in density with firing temperature.
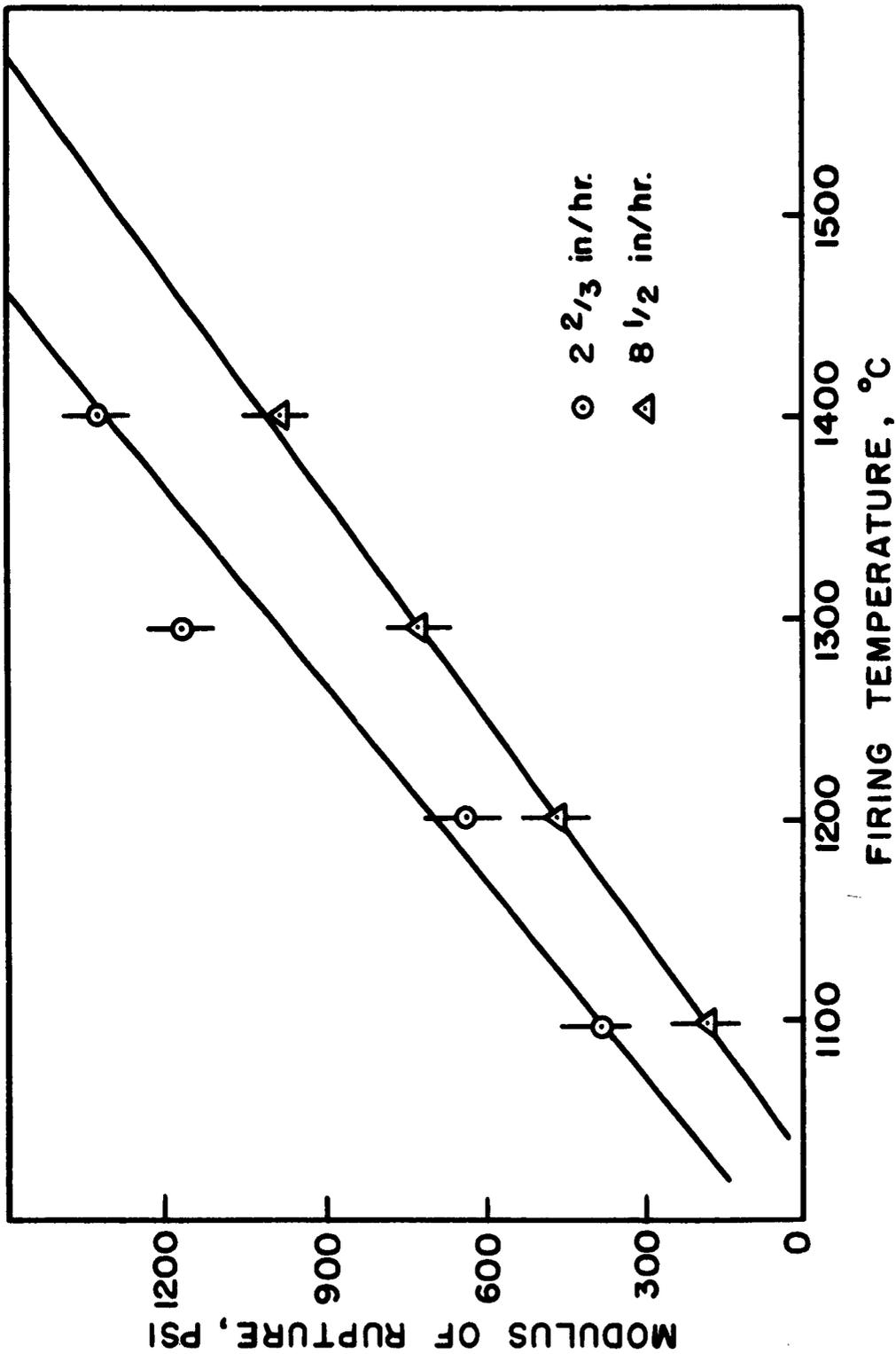
Figure 13 - Modulus of rupture vs. firing temperature.

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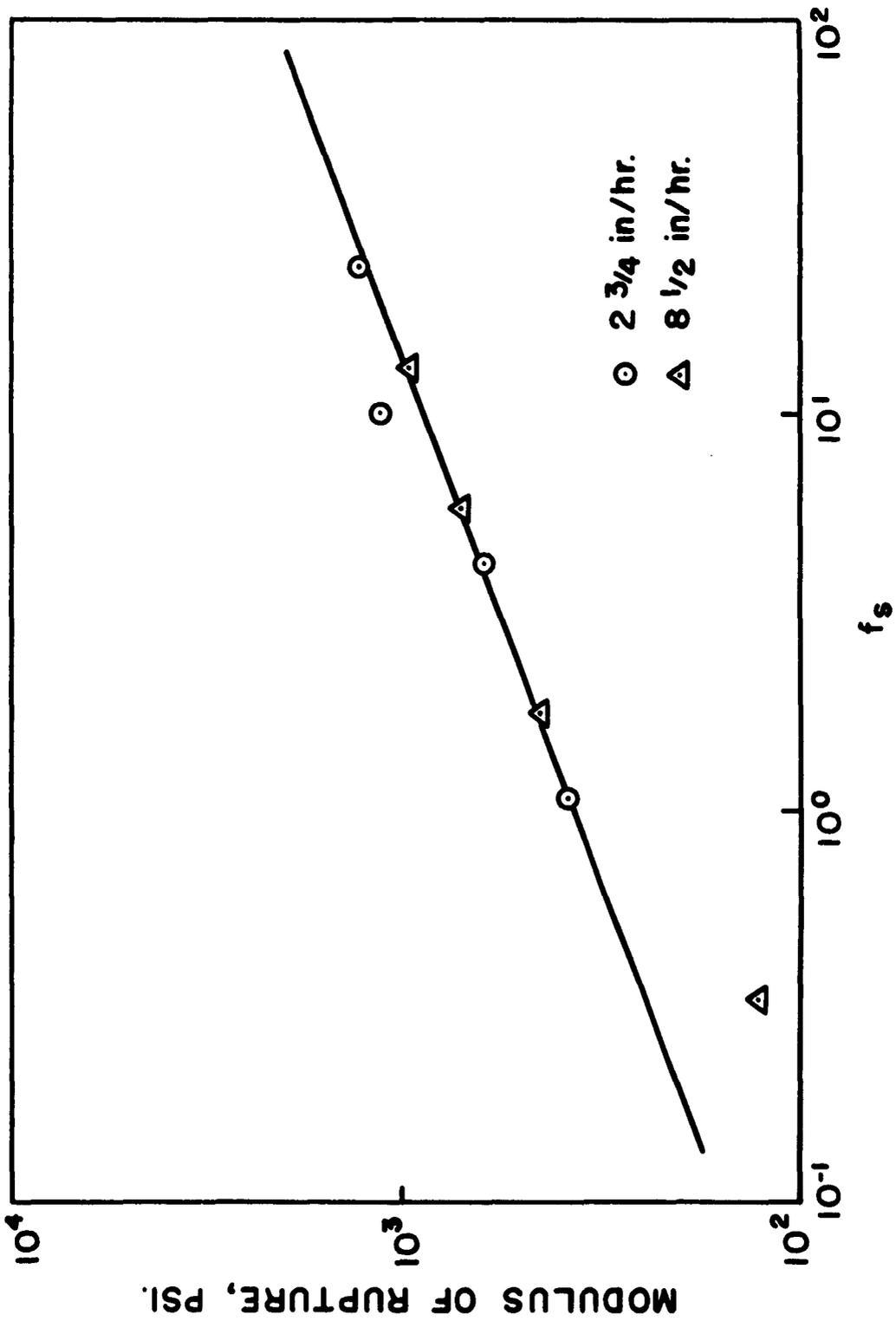
Figure 14 - Modulus of rupture vs. relative amount of spinel formed.

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Aeronautical Systems Division, Dir/Materials and Processes, Metals and Ceramics Lab., Wright-Patterson AFB, Ohio.
Rpt No. ASD-TDR-62-732. A FUNDAMENTAL STUDY OF THE VARIABLES ASSOCIATED WITH THE MIXING OF CERAMIC RAW MATERIALS. Final report, Apr 63, 24p. incl illus. and refs.

Unclassified Report

This summary report presents the progress completed on the investigation of the mixing and sintering characteristics of Al_2O_3 and MgO particles. Separate inter-related areas of research have been investigated which include particle preparation, dry and wet mixing, and the sintering behavior of Al_2O_3 and MgO.

The techniques of particle reduction and analysis of particle size distribution have been investigated by determining the variables which affect the

(over)

1. Ceramic materials
2. Sintering
3. Particles
I. AFSC Project 7350 Task 735001
II. Contract AF 33(616)-7763
III. University of Calif., Berkeley, California
IV. D. W. Fuerstenau, et al
V. Aval fr OTS
VI. In ASTIA collection

resulting particles. Emphasis has been placed on control conditions for a jet pulverizer.

Variables encountered in the dry mixing operation have been found to be the mixer design, loading methods, and sampling methods. Analysis of these variables has been made by determining the standard deviation from a statistical number of samples. Surface properties of Al_2O_3 and MgO in aqueous media have been investigated in order to understand wet mixing behavior. Determinations of the nature of the electrical double layer of the particles have been made.

The basic mechanisms and the characteristics of the reaction between MgO and Al_2O_3 have been studied with single crystals. The understanding of the fundamentals of the reaction has enabled the establishment of optimum sintering conditions. Some of the properties of sintered compacts due to varied sintering conditions have been determined.